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DIELECTRIC RADIO FREQUENCY HEATING OF PROPELLANTS INCIDENT INVESTIGATION AND APPLICATIONS

by

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ABSTRACT

Double-base propellant is heated to ease extrusion by utilizing the propellant dielectric properties in a radio-frequency oscillatory circuit. This paper presents information developed during investigation of a series of fires which were found to be related to the heating methodology and dielectric properties of the propellant. The content of this paper was extracted from reports and memorandum prepared by the incident investigation team of Chester E. Davis, E. Gordon Powell and the author. That the first two members were the major contributors to the investigation team is acknowledged.

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Double-base propellants are extruded to form rocket motor grains. Hydraulic presses and dies are utilized and the process is well documented. To ease extrusion, the propellant is softened by heating, normally to 130-140°F. Heating is accomplished basically in two ways; oven heating or radio-frequency heating utilizing the dielectric properties of the propellant. Problems encountered and information developed while utilizing dielectric heating at the Naval Ordnance Station are summarized in this discussion.

To assure understanding, a brief familiarization with the double base propellant process is in order. In this process, the ingredients, nitrocellulose, nitroglycerin, plasticizers and burning rate modifiers are mixed together in a water damp paste. This paste is put through heated rollers to drive off the water and give desired physical properties. The product of these rolling mills is a sheet, nominally 0.080 inch thick. These sheets are cut into strips 4 inches wide which are rolled into right circular cylinders called carpet rolls (nominally 15 inch diameter). The carpet rolls are heated and placed in the extrusion press for forming the propellant into rocket motor grains. The two principal means of heating the propellant carpet rolls are thermal ovens and dielectric heaters.

The thermal method of heating requires the carpet rolls be placed in an oven and allowed to come to a uniform temperature throughout. Because the carpet roll is fairly thick, a considerable period of time is required to attain desired temperature in the center. Also the thermal oven must be set near the final temperature desired for the propellant and this reduces thermal force and results in long heating periods. For these reasons, the thermal ovens are known as "soaking" ovens. Problems associated with soaking ovens are the investment in ovens required to support even a modest production capability and the effects of extended thermal soaking on the propellant. Of the latter, the major effects are volatilization of plasticizer and hardening of the propellant. Consequences are process and quality problems in the extrusion of carpet rolls to form propellant grains.

The second method of heating, that is dielectric heating, applies a high or radio-frequency electric field to carpet rolls situated as the dielectric in a parallel plate capacitor as shown in Figure 1. This is basically a resonant capacitive-inductive circuit. The electric field takes effect throughout the propellant thus eliminating thermal diffusivity as a factor. Heating time is reduced from ~24 hours for soaking ovens to ~20 minutes.

The phenomena involved in dielectric heating can be simplified to the following concepts:[1]

a. The electric field potential gradient causes distortion and orientation of atoms and molecules by displacement of electrons with respect to the nucleus;

b. Both polar and non-polar molecules are affected;

c. The effect of the displacement is to reduce the field gradient within the dielectric material.

These concepts are illustrated in Figures 2 and 3.

To digress a bit, note that both metallic conductors and dielectric or nonconductors can be heated by radio-frequency fields. The phenomena involved are different but pertinent to some of the events to be discussed. In heating conductive (metallic) materials, the imposed electric field induces motion of free electrons. Resistance to their motion by the atomic matrix results in heat generation. This is known as inductive heating and has extensive application.[2] Suffice for this discussion to visualize conditions occurring within a conductor located in an electric field; the free electrons concentrate at extremities and negate the field within the conductor thus creating concentrated charges and high electrical potentials.[1]

Returning to dielectric materials which have few free electrons, the distortion and displacement of atomic and molecular charges and resistance of the material matrix to orientation generates heat. As these phenomena occur throughout the material, heating does also.

An aid to visualizing the properties of the material in an electric field is to determine an equivalent circuit for the material. The basic circuit illustrated by Figure 4 shows that if a field is applied across a cube of material, the admittance (reciprocal of impedance) has both a capacitive or susceptive component and a conductive component. The conductive/resistance component is representative of metallic response; susceptive/capacitive component is representative of dielectric response to imposed fields. Note that when high frequency changes are made in the field, the molecules and electrons do not have time to achieve equilibrium with the field. This creates conditions such as anomalous dielectric dispersion.[3]

The admittance of RF electrical energy into a material equivalent circuit can only be described in mathematical terms by complex number notation.[2][3][4] This notation utilizes real and imaginary components. Application includes real and imaginary terms in the power factor, a concept involved in supplying energy to a dielectric in a resonant capacitive-inductive circuit and in other alternating current electrical circuits. In vector notation, the angle between vectors representing capacitor charging current and total current is the loss angle. The loss tangent or dissipation factor is the ratio of loss current to charging current.

The preceeding remarks were intended to give a brief familiarization with the nomenclature of extruded double-base propellant processing, RF heating, and dielectric material phenomena. More detailed discussion can be found in references 1 thru 4 and any good text on high frequency electrical circuits.

There are various conditions inherent in dielectric heating which should be mentioned. Some that are important but not well characterized are changes which occur in the materials conductivity, dielectric constantand power factor or loss tangent as the frequency and material temperature vary. For example, as the material heats up, its electrical parameters change which cause change in the resonant frequency of the circuit. The dielectric constant, power factor and conductivity all vary with frequency. Also, it is important to note that a general property of dielectrics is for the imaginary part of the dielectric constant to increase with temperature. These interactions can lead to properties changes causing frequency shift toward better coupling and greater energy absorption by the dielectric. Sometimes these can cause problems in controlling heating of propellant.

Another condition which occurs is creation of standing waves, i.e., non-uniform voltage distribution. This is a function of electrode dimensions and wave length. Standing waves can be tuned out, however, they and the material characteristics previously mentioned can generate significant potential gradients within the material.

At Naval Ordnance Station, Indian Head, RF dielectric heaters have been utilized for heating propellant carpet rolls. The propellant enters the circuit as the dielectric in a parallel plate capacitor arrangement as illustrated in Figure 1B. Note that Figure 1A shows insulating rubber pads which separate the propellant from all metal surfaces. This eliminates direct application of high electrical potential from a conductor to the propellant. The pads are of low-loss dielectric material.

During the period 1950 to 1973, ten fires had occurred in the high frequency heaters. In most of these, foreign material or defects in the insulating cover of the electrode plates were considered as the most likely cause. For example, a metal stem thermometer for determining propellant temperature, if left in the carpet rolls during heating, can cause localized heating. The metal conducts in the applied electric field and concentrates the field at edges and points. This concentrated field causes localized heating in the propellant and could result in an arc. As these would occur within the carpet roll where the thermometer is utilized, ignition is a possiblity. Other possible causes are water which is a highly polar molecule that may lead to localized concentrated fields and corona and arc discharge from various parts of the heater. Minor amounts of water are considered likely to evaporate before ignition temperature is attained. Corona discharge and arcs have been observed however they consistently are located on parts of the heater remote from the propellant.

In 1973, and early 1974, a series of five fires occurred with one particular propellant. As the investigations progressed from one fire to another, the obvious foreign item causes were ruled out. This led to the conclusion that the ignition cause was involved in heater operation and propellant proper-

ties. Following paragraphs discuss items considered and action taken without regard to chronology except that intensive effort was initated after the fourth fire and the fifth fire occurred during this time.

Note that this was the first aluminized propellant subjected to dielectric heating. Limited tests had indicated that aluminized propellant could be heated safely. Over 1 1/4 million pounds had been heated prior to the first fire so aluminum can not be considered the sole cause of ignitions. X-ray examination of carpet rolls from the lot involved in the last fire showed areas $\sim$1/16 inch diameter with increased attenuation. Visual examination did not reveal any cause for the attenuation and the propellant was subsequently dielectrically heated and extruded without incident.

The possibility that corona discharge or arc discharge was the ignition source was considered. Operating personnel had occassionally observed corona discharge during normal operation of the heaters. However, the corona was always observed on parts of the heater some distance from the propellant. One function of the insulating pads is to smooth the interface between propellant and electrodes and eliminate sharp points which are likely sources of discharge. Also, tests indicated the propellant in question would withstand current densities substantially in excess of typical corona discharge for a time longer than the cycle time of the heater. Hence corona discharge was considered an unlikely cause but the sharp edges of conductors were blunted to eliminate high field potential points that cause discharge.

Arc discharges were considered a less probable cause than corona as none had been seen or heard and plate current meters had not indicated erratic fluctuations typical of arc discharges. Tests indicated arc discharge current density was capable of igniting the propellant. Regardless, the absence of evidence of arc discharge under any condition of heater operation led to the conclusion that they were not the cause of the fires.

Foreign material in the propellant or facility was considered. Metallic foreign material discussed previously in regard to fires prior to 1973, was ruled out for lack of evidence. Also, experienced operators were in charge and thoroughly inspecting for foreign material. Dielectric foreign material was determined unlikely after determining the dielectric constant of the propellant as few materials have a constant with an imaginary part which exceeds that of the propellant. Ordinary water is one but was ruled out as known not to be present in most of the fires. In any case, minor amounts of water would tend to vaporize before ignition temperature is reached.

Possible erratic problems with the heater operations were considered. Manufacturer service personnel inspected the equipment following one of the fires. In this effort, the RF voltage sensor was relocated from the power supply to the heater plates in order to measure the voltage impressed across the propellant.

Parallel measurements across the propellant and the generator output were taken during the change. As expected, the voltage across the capacitor (heater plates) in the resonant inductive-capacitive series circuit was higher than the voltage across the total circuit measured at the generator output. Based on these limited results, the voltage across the propellant was reduced by retuning the RF generator. Output voltage was reduced from 4-5 kilovolt to 1.5-2 kilovolts.

During the investigation following the fifth fire, operators mentioned that the plate current meter readings were always higher with the aluminized propellant. Also, there had been a gradual increase in heating rates in the interval between fires. Periodically control adjustments had been made to keep within the 3 1/2^{o}F/munute heat rate. A specific cause of the increase in heating rates was not determined. Two possibilities are a gradual change in propellant properties or drifting of the heater electronics. The first might explain the onset of fires after more than a million pounds of incident free operations. The second, drifting of heater electronics, had not been a detectable phenomena in prior operations with any propellant. Drifting may have occurred by some subtle feedback mechanism. This conjecture is based on the subsequent finding that the heaters having fires were tuned such that as the propellant heated, the change in dielectric properties caused the RF generator frequency to shift toward better resonant coupling. This produces higher voltage across the propellant which in turn causes higher heating rates, a feedback situation. If there was initially any localized inhomogenity in the propellant in either temperature or dielectric properties, the feedback could result in a localized thermal runaway situation. The higher the temperature in the inhomogeneous element, the faster it heats. If the localized heating rate exceeds the thermal diffusivity then ignition temperature can be reached. Unfortunately, no localized inhomogenities could be found though this was not considered as confirming their absence.

Eventually, the dielectric properties of the aluminized propellant NOSIH-AA-6, were hypothesized to be directly involved in causing fires. An investigation of these properties was made in comparison to N-5 propellant which had extensive history without fires. Tests were also made on a "non-hazardous" dummy propellant of nitrocellulose, dibutyl phthalate and aluminum that is sometimes used to check extrusion processing. Results were that the real part of the dielectric constants of AA-6 and N-5 were about the same (approximately 10). As hypothesized, AA-6 showed an imaginary part of the dielectric constant about twice that of N-5. The dummy propellant had a real dielectric constant lower than N-5 or AA-6 but the imaginary part greater than that of AA-6. (This dummy propellant should not be used to check out dielectric heaters as it will burn.) As the power factor or energy absorption is directly proportional to the imaginary part of the dielectric constant, it follows that AA-6 heats faster than N-5 for the same heater conditions.

To confirm the laboratory findings, full-scale tests were made. These measured the dielectric properties of N-5 and AA-6 as normally loaded into the heaters. Low power lab equipment was connected in place of the RF power gene-

rator to preclude actual heating of the propellant. The measurements were not entirely reliable because of poorly known transmission line effects, however, it was apparent that AA-6 had a significantly higher imaginary dielectric constant. These tests gave a rough indication of change in dielectric properties as a function of frequency. Also, by using propellant conditioned to different temperatures, frequency variation with temperature was briefly studied.

Measurements were made of the tuning of each of the three operational heaters. The oven which had not experienced any fire with the AA-6 propellant was found to be tuned somewhat differently from the others but no firm conclusion could be established as that oven was seldom utilized for AA-6 propellant processing.

All of the evidence indicated that the problem had been outlined sufficiently to attempt a solution. The obvious approach was to change the RF generator tuning so that the heating rate was less. Initially, it was planned to achieve this by tuning such that the changing properties of the propellant would pull the load circuit away from resonance with the generator as the propellant temperature increased. After other adjustments, this detuning was achieved.

Lowering of the heating rate was attained in trials but not as much as desired for safe operation. Adjustment of the normal controls made little progress toward lowering the heating rate. A study of the generator circuit determined that changing the tap on the output RF transformer was a modification that would allow improved control range. The tap was changed to a lower position with respect to ground which had the effect of lowering power input to the load. Controls then functioned normally to adjust the heating rate to 3.5°F/minute with AA-6 propellant. Rate with other propellants was lower.

As the fires had occurred early in the heating cycle, it was considered that initial propellant temperature condition was a factor. To even out any inhomogenities, a split heating cycle was utilized. This cycle applied heat, then a rest period of a few minutes before heating again. This inefficient technique was no longer required after the heaters were improved by the addition of automatic load controls.

With the heating rate dependent on propellant properties, control settings had to be changed with each propellant. This was achieved by manually relocating (by cranking mechanism) the output tap on the plate current load coil. Note must be made that for all propellants, the plate current and consequently the heating rate changed as the powder temperature changed. With the ovens tuned to decrease coupling as the propellant temperature increased, the plate current and heating rate decrease with time in the heating cycle.

This discontinuous and decreasing rate heating cycle and need for changing

control settings for different propellants adversely affected operations. Therefore, efforts were made to determine modifications to equipment that would return to continuous heating cycles without causing fires. This was accomplished by noting that the plate current load coil, normally set at a point by manually cranking, was amenable to controlled positioning. Coupling this positioning with the tuning to pull the generator away from resonance as the propellant heats up appeared to provide a means for safe and efficient operation. To understand this, it had to be noted that for fixed control settings the initial heating rate would be near the maximum of 3.5°F/min and would decrease with cycle time and increase in propellant temperature to <2°F/min at cycle end.

Automatic load controls were installed which controlled heating rate by changing the position of the plate current load coil output tap. The controls were programmed to automatically position the tap at the low current control set point at the end of each cycle. This was intended to assure that initial heating of each propellant charge was at low power thus reducing the effects of any inhomogenities and reducing chances for ignition which normally occurs early in the heating cycle. As the propellant heats up and internal conditions become uniform, the controls change tap position to increase plate current to a set level and thus apply more power to the propellant. Since the heaters are tuned to pull away from resonance as the propellant increases in temperature, balancing plate current increase/maintenance to set point against resonance detuning allows achieving a uniform heating rate through the cycle. It was found that with these controls, maximum heating rate could be reduced within the time alloted for heating. This was considered an improvement in safety.

Because there are individual system differences, each of the three heaters was characterized for plate current versus heating rate. The automatic load controllers were adjusted to start at a minimum setting below the operating level. As the cycle progresses, plate current output coil tap is automatically repositioned to increase current to attain and maintain the operating level. The improved heaters have been operated without a fire since 1974.

There still remains one unanswered question, what change occurred to trigger a series of fires after initially processing 1 1/4 million pounds without incident. Admittedly, data generated in the investigations revealed that operation with AA-6 must have been closer to ignition conditions than is the case with prior propellants processed.

Furthermore, the fact that two heaters were involved in fires would indicate that drifting of the RF generator and circuits was not the sole cause. Duplicate failure modes in two separate systems at nearly the same time is a low probability occurrence. This heater drift rationale has one weakness, and that is maintenance and adjustment, which, consistently performed in one direction on both heaters, may have eventually shifted them from no-fire to fire condition. This could not be determined and is thought unlikely based on operating and maintenance records and personnel memories.

Subsequent to the last fire, another aluminized propellant (2 1/2 Al) has been processed successfully although it is probably more sensitive than AA-6 to the power loading rate. This conjuncture is based on limited observation of the plate current when heating cycles begin for the two different propellants.

Only one item is left, the propellant. Some subtle change in propellant ingredient or processing changed the dielectric properties. Conjecture would be that the change was in the plasticizers as they generally have greater dielectric properties change with frequency and temperature than the other materials. This is supported by the observation that the second aluminized propellant with half the aluminum content of AA-6 appears more sensitive and has a higher plasticizer content. (Note that Nitroglycerin, a major ingredient has a real dielectric constant of 19.)[5] The same difference would tend to rule against aluminum as the culprit. Although processing resulting in concentrations of aluminum could result in a tendency toward fires, it is more likely that aluminum (less than 5%) is an accomplice rather than the culprit. Further studies of the dielectric nature of propellants would be interesting but not necessary items and are not planned as current operations are satisfactory.

Reference:

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3. SCIENTIFIC ENCYCLOPEDIA, Van Nostrand, 5th Edition
4. COMPLEX VARIABLES AND APPLICATIONS, R. V. Churchill, McGraw-Hill
5. CHEMISTRY AND TECHNOLOGY OF EXPLOSIVES, Vol. 2, T. Urbanski, Pergamon Press

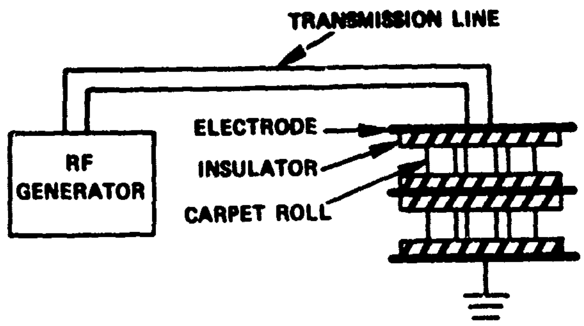


FIGURE 1A. DIELECTRIC OVEN

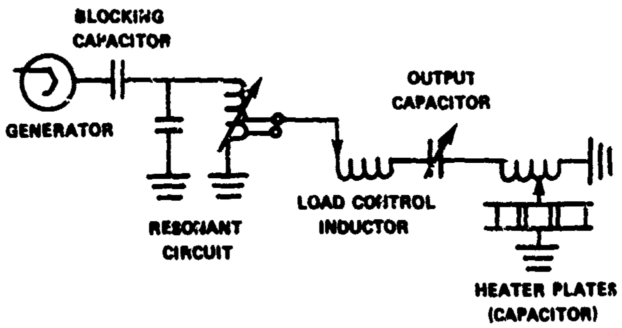


FIGURE 1B. EQUIVALENT CIRCUIT OF RF DIELECTRIC HEATER

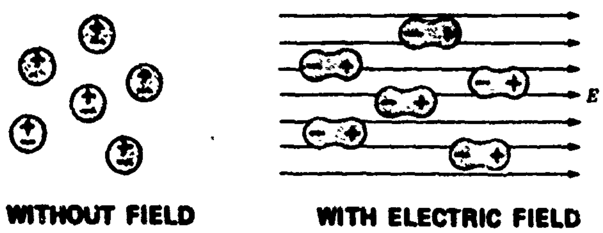


FIGURE 2A. BEHAVIOR OF NONPOLAR MOLECULES IN THE ABSENCE AND IN THE PRESENCE OF AN ELECTRIC FIELD (From Sears – University Physics)

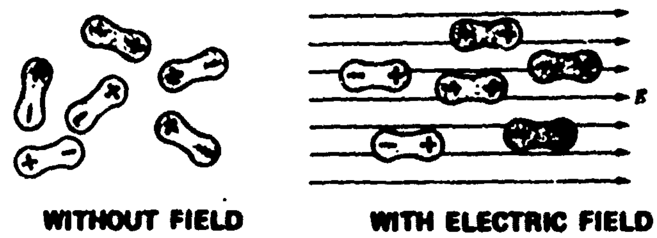


FIGURE 2B. BEHAVIOR OF POLAR MOLECULES IN THE ABSENCE AND IN THE PRESENCE OF AN ELECTRIC FIELD
(From Sears – University Physics)

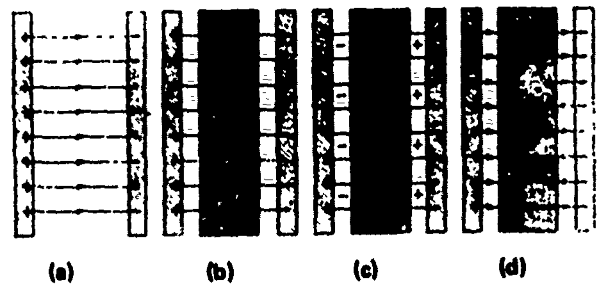


FIGURE 3. (a) ELECTRIC FIELD BETWEEN TWO CHARGED PLATES. (b) INTRODUCTION OF A DIELECTRIC. (c) INDUCED SURFACE CHARGES AND THEIR FIELD. (d) RESULTANT FIELD WHEN A DIELECTRIC IS BETWEEN CHARGED PLATES.
(From Sears – University Physics)

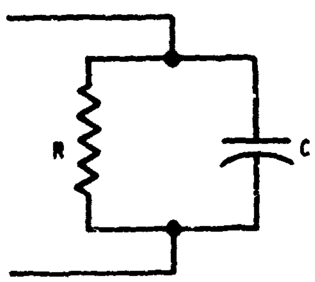
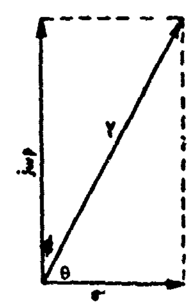


FIGURE 4A. EQUIVALENT CIRCUIT FOR A CUBE OF MATERIAL
(From Brown, – Radiofrequency Heating)



$Y = \frac{1}{R} + jwc$

or alternately

$Y = \sigma + jwp$

Power Factor or loss angle. $= \frac{\sigma}{wp} = \tan \phi$

FIGURE 4B. ADMITTANCE DIAGRAM OF A COMPLEX CAPACITOR
(From Brown – Radiofrequency Heating)